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Mechanical and Plastic Properties of Elements Made of Steel X5CrNiCuNb16-4 Using the Selective Laser Melting Technique (SLM)

Abstract: The article presents results of tests performed using an MCP HEK Realizer II system applied in the selective laser melting (SLM) of metallic powders. Specimens subjected to the SLM process were made of powder, the chemical composition of which corresponded to that of solid steel X5CrNiCuNb16-4. The material was subjected to mechanical tests (concerning tensile and impact strength) and compared with the properties of the solid steel. The research-related tests also involved microstructural observations involving the use of a Neophot 32 metallographic microscope (Zeiss) and fractographic analysis. The tests revealed that the mechanical properties of the printed material subjected to the SLM process were lower ($R_{0.2}$ by 45% and R_m by 35%) than those of the solid material and were determined by the properties of the metallic matrix and the porosity of the printed element, the average value of which amounted to 3%. The mechanical properties of the printed material were also significantly affected by the direction of the external load in relation to the orientation of the deposited layers of the material (which was demonstrated during impact bend tests). The summary contains the assessment of the tests and the presentation of advantages resulting from the application of the new technology enabling the volumetric consolidation of metallic powder.

Keywords: SLM process, metallic powders, tensile, impact strength, stainless steel

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Introduction

Technological processes enabling the additive manufacturing of parts and elements has been developed since the early 1990s [1]. The first one of such processes was stereolithography (SLA), developed as early as in the 1980s. Presently, selective laser sintering (SLS) and selective laser melting (SLM) techniques belong to the fastest and most intensively developing manufacturing processes. Both methods (SLS/SLM) involve the fabrication of elements through the laminar joining of powdered materials using the laser beam. The technology provides enormous possibilities by enabling the very fast fabrication of precise and homogenous elements made of various metallic materials, including stainless steels, tools steels as well as aluminium, titanium, nickel, cobalt, chromium and magnesium alloys [2–5]. Models are usually used as prototypes making it possible to assess the functionality or correctness of designs. Over the past years, the technology has become the subject of

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interest in the automotive, aviation and space industries as well as in medicine or tool fabrication [6–9], where it is increasingly commonly used to make finished products.

In Poland, in spite of entrepreneurs' limited trust and relatively high costs, the SLM technology has been successfully used in prosthetics, implantology, aviation, prototyping, carmaking or injection mould production [10–12]. However, when analysing the interest in the fabrication of metallic products using additive manufacturing methods, rapid technological development, the wide spectrum or materials and numerous tests confirming the effectiveness of the method enabling the obtainment of elements characterised by very good mechanical parameters, it is expected that the method will enjoy significantly growing popularity in the years to come. The primary advantage of the method is the high rate at which precise elements of complex geometry are manufactured.

In terms of its operating principle, the SLM technology is composed of three stages:

Providing a protective atmosphere in the working chamber by using inter gas shielding metallic powder against oxidation and increasing temperature inside the chamber.

The deposition of a thin layer of metallic powder by a crusher onto the working platform, followed by the selective melting of metallic powder particles by the laser beam.

Lowering the working platform by a value corresponding to the thickness of a single layer (each time) after melting metallic powder particles until the obtainment of the entire geometry of a given element.

The publication presents results of tests performed in the rapid prototyping laboratory of the Faculty of Mechanical Engineering and Mechatronics of the West Pomeranian University of Technology in Szczecin, being in possession of an MCP HEK Realizer II machine, enabling the selective laser melting (SLM) of metallic powders.

Testing methodology

The specimens used in the tests were made using the SLM technology and powder, the chemical composition of which corresponded to that of steel X5CrNiCuNb16-4. The process parameters included a laser power of 100 W, a laser beam travel rate of 250 mm/s and a powder layer thickness of 0.05 mm.

Tables 1 and 2 present the chemical composition of a specimen made of steel X5CrNiCuNb16-4 and specimens subjected to laser sintering followed by stress relief annealing.

Steel X5CrNiCuNb16-4 in the solid state is one of the most commonly used stainless chromium-nickel precipitation-hardened martensitic steel grades with an addition of copper. The steel is characterised by high corrosion resistance and good mechanical properties (including hardness).

The specimens made using the SLM method were subjected to stress relief annealing. The performance of such a procedure is consistent with recommendations formulated by the manufacturer of the powder. As can be found in reference publications [13–15], standard heat treatment methods, used successfully in cases of solid elements, are not optimum for elements made using the SLM method.

Mechanical properties of the test materials were identified in tensile strength and impact strength tests. The static tensile test was performed using specimens having a square cross-section (10 mm × 10 mm) and measurement length l_0 = 50 mm. The impact strength test was performed using specimens having standard dimensions (10 mm × 10 mm × 55 mm) with a 2 mm deep V-notch. The study also involved the performance of microstructural observations using a Neophot 32 metallographic microscope (Zeiss) and Beraha's I reagent (Fig. 5, 6) as well as the assessment of fractures.

Table 1. Chemical composition of steel X5CrNiCuNb16-4

Chemical composition [%]										
C	Si	Mn	P	S	Cr	Mo	Ni	Cu	Al	Nb
0.039	0.184	0.901	0.024	0.0068	15.35	0.091	4.93	4.082	0.026	0.241

Table 2. Chemical composition of the specimen after SLM and stress relief annealing

Chemical composition [%]										
C	Si	Mn	P	S	Cr	Mo	Ni	Cu	Al	Nb
0.052	0.605	0.832	0.017	0.0098	15.72	0.209	4.956	5.076	0.008	0.264

Test results

The comparison of the properties and effectiveness of the SLM method necessitated also the performance of tests concerning the initial material, i.e. steel X5CrNiCuNb16-4 in the solid state. The results of the tests related to mechanical and plastic properties are presented in Figures 1 and 2.

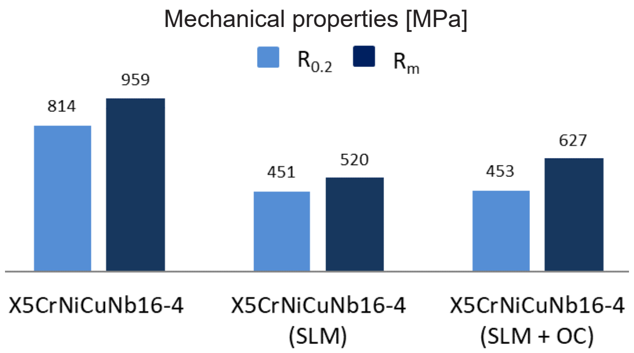


Fig. 1. Average values of strength properties determined in the static tensile tests involving the specimens made of steel X5CrNiCuNb16-4 and the specimens subjected to selective laser melting (SLM) and those subjected to both selective laser melting and stress relief annealing (SLM + HT) – heat treatment

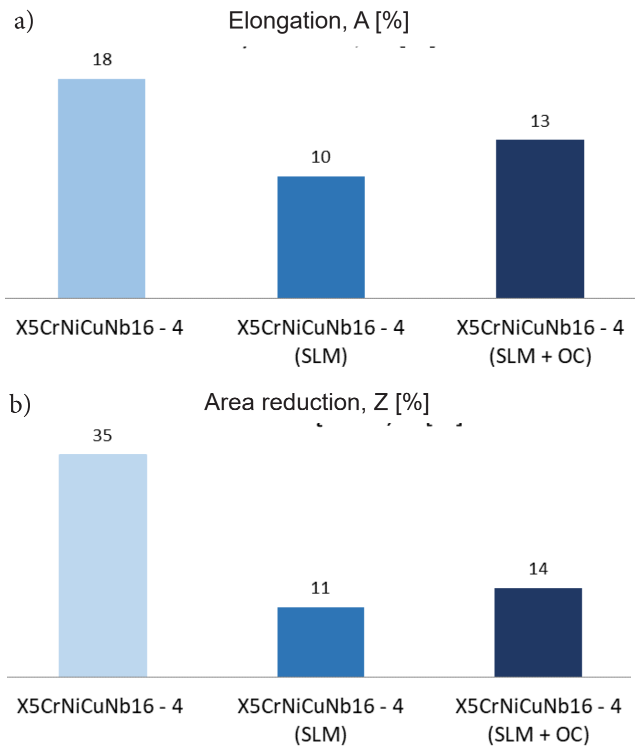


Fig. 2. Average values of plastic properties determined in the static tensile test: a) elongation and b) area reduction

The preparation of the specimens for impact strength tests involved taking into account an additional variable factor, i.e. the direction in which the specimen was cut out in relation to the direction of layer build-up. Figure 3 presents three positions of the V-notch (having an angle of 45° and a depth of 2 mm) in relation to the direction of the build-up of material layers.

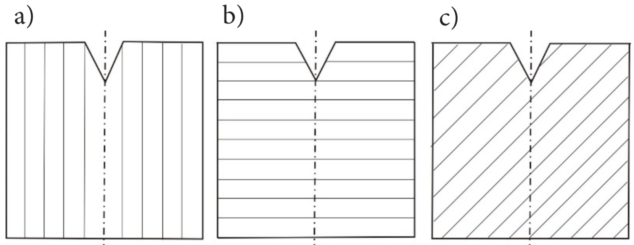


Fig. 3. Position of the notch axis in relation to the direction of the build-up of individual layers of the material: a) notch situated in parallel, b) notch situated perpendicularly and c) notch inclined at an angle of 45° in relation to the direction of layer build-up

Figure 4 presents results obtained in the impact strength tests involving the specimens made of the solid steel and those made using the SLM method. The tests involved the use of the Charpy-V method performed at a temperature of +20 and that of –20°C.

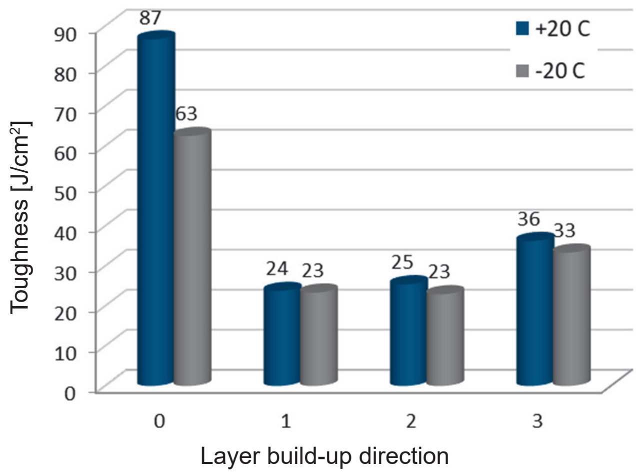


Fig. 4. Average impact strength values at two testing temperatures: 0 – solid material; 1, 2, 3 – specimens made using the SLM (additive manufacturing) method: 1) notch situated in parallel to the layer build-up direction, 2) notch situated perpendicularly and 3) notch inclined at an angle of 45°

The results of the microstructural tests of the standard specimen made of the solid steel and those related to the specimen obtained after selective laser melting are presented in Figures 5 and 6.

Discussion

The high similarity of structural features of materials obtained using the SLM method and metallic sinters obtained using conventional methods of powder metallurgy justifies (with high likelihood) the statement that the basic structural feature of these materials which determines their mechanical

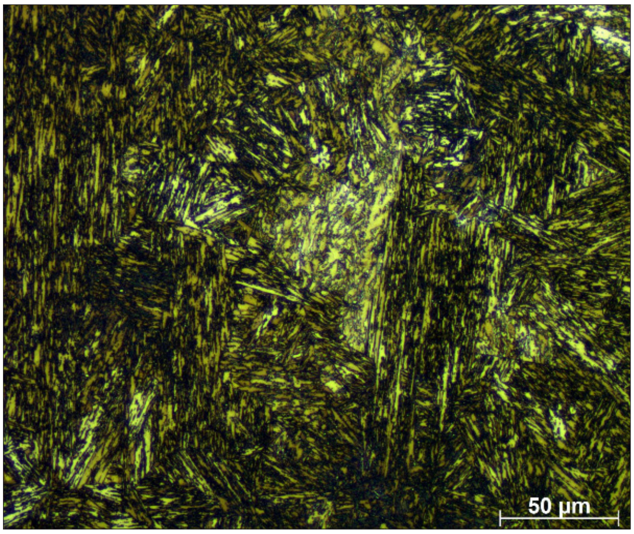
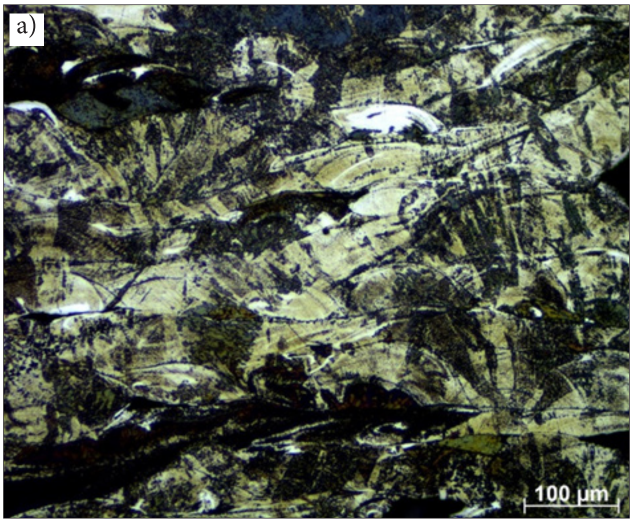


Fig. 5. Martensitic structure of solid steel X5CrNiCuNb16-4 (etchant: Beraha's I reagent)



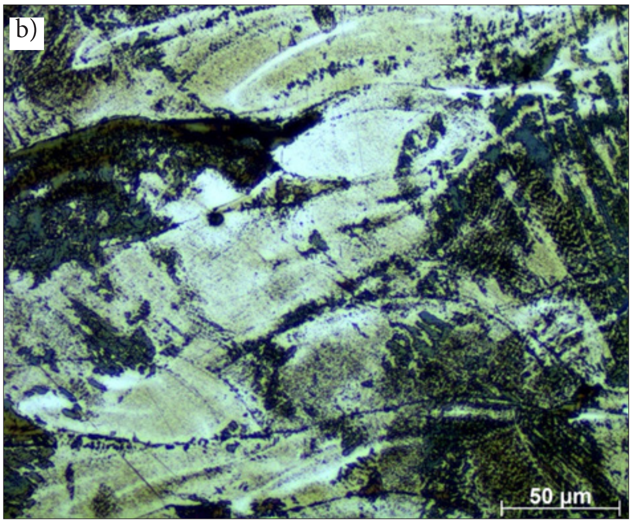


Fig. 6. Layers in the specimen subjected to selective laser melting (etchant: Beraha's I reagent): a) dual-phase ferritic--austenitic structure (duplex) and b) view of precipitated carbides of alloying elements (Ni and Cr)

properties is porosity. This issue is discussed in numerous publications related to powder metallurgy. Mechanical and plastic properties of sinters are determined by properties of the metallic matrix and, to a similar extent, by porosity. The sinter, defined as a porous medium, is composed of the solid material (constituting the matrix) and pores (cavities) situated within the entire volume. In terms of the porous medium, there is certain small volume V_0, which is equated with a particle of the solid medium. All parameters characterising the medium lose their meaning in relation to volume smaller than V_0, in accordance with the following dependence:

$$V_{1p} << V_0 << V \qquad (1)$$

where
V_{1p} – volume of a single pore,
V – volume of the medium.

The density of centre ρ and porosity Θ are defined as follows:

$$\rho = \lim_{\Delta V \Rightarrow V_0} \frac{\Delta M}{\Delta V} \qquad \Theta = \lim_{\Delta V \Rightarrow V_0} \frac{V_p}{\Delta V} \qquad (2)$$

where
ΔM – mass of the material around a point under consideration,
ΔV – volume of the medium,
V_p – total volume of pores contained in volume ΔV.

If pores do not contain any mass, the density of medium ρ is defined as follows:

$$\rho = \rho_L (1 - \Theta) \qquad (3),$$

where
ρ_L – density of the solid material,
Θ – porosity.

The relative density of medium ρ^* is determined using the following dependence:

$$\rho^* = \frac{\rho}{\rho_L} = 1 - \Theta \qquad (4)$$

Based on equation (4), the tests involved the determination of the theoretical density of the material obtained using the additive manufacturing method (SLM) and led to the identification of the average value of relative density amounting to 0.97 (97%). Hence, the average porosity of the selective laser melted specimens was Θ = 3%. It was also observed that the porosity of specimens depended slightly on the material layer build-up direction.

The results of the tests concerning the specimens made using the MCP HEK Realizer II machine and powdered material X5CrNiCuNb16-4 justified

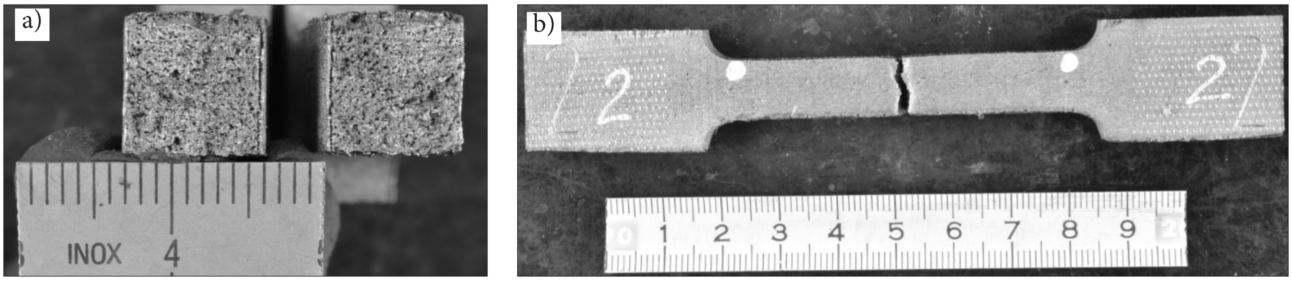


Fig. 7. Specimen obtained using the additive manufacturing method after the tensile test: a) fracture and b) location of the fracture in relation to the main axis of the specimen

the conclusion that the specimen obtained using the additive manufacturing method was characterised by significantly lower mechanical properties, i.e. $R_{0.2}$ was by 45% lower and R_m was by 35% lower than those of the solid material (Fig. 1). Similarly, the parameters of material plasticity, i.e. elongation A and area reduction Z, were significantly lower in the additive manufactured specimens (Fig. 2). During the tensile test, the specimen elongated uniformly along the length of the measurement part. After exceeding the maximum value of tensile force, the specimen did not contain the neck (the location of the deformation). The fracture was oriented perpendicularly in relation to the highest tensile stress (axial), was preceded by slight plastic strains and had the form of dividing brittle fracture (Fig. 7).

The particularly significant effect of porosity, leading to the reduction of mechanical properties was observed during impact bend tests (Fig. 4). The tests revealed that toughness was significantly affected by the direction of layer deposition in relation to the direction of forces. The highest toughness values were observed in the specimens with the notch situated diagonally in relation to the deposited layers. In the specimens with the notch situated in parallel and perpendicularly in the laser build-up direction, the obtained values of toughness were significantly lower but similar. Such a situation was connected with the direction of the effect of maximum tensile stresses present at the bottom of the notch in relation to the direction of material layer build-up. The foregoing justified the conclusion that the toughness of the materials made using the SLM method was the resultant of the mechanical properties of the matrix, the porosity of the material and the orientation of maximum tensile stresses in relation to the direction of material layer build-up.

Maximum tensile stress σ_{max} present at the bottom of the notch could be determined using the following equation:

$$\sigma_{\max} = 2\sigma\sqrt{\frac{\Delta H}{r}}, \text{ MPa} \qquad (5)$$

where
- σ – tensile stress without the concentration of stresses, MPa
- ΔH– notch depth, mm
- r – notch bottom radius; in relation to the V-notch it was assumed that $r = 0.1$ mm – 0.2 mm

The analysis of equation (5) led to the conclusion that toughness results depended both on the position of the notch axis in relation to the layer build-up direction and, which was equally important, on the position of the notch bottom surface in relation to the deposited layers. If the bottom of the notch, determined by notch depth parameter ΔH, was located within the layer boundary area, maximum tensile stress $\sigma_{max,}$ initiating the cracking of material would be lower, which, in turn, would affect toughness results. Also the manner and precision used when making the notch by applying removal machining in the laser-melted material proved to have a greater effect on the measurement result than that observed in relation to the solid material.

Figure 8 presents the arrangement and orientation of tensile stresses triggered by the notch effect in relation to the material layer build-up direction. When the direction of tensile stress effect was perpendicular or parallel to the layer build-up direction, the material ruptured on the borders of layer (being structurally the weakest area of the additive manufactured specimen (Fig. 8a, b). In the situation as presented in Figure 8a, the tensile stress-triggered strain was located in the area of layer boundaries and induced the cracking of the laser-melted specimen. In terms of the direction of tensile stress consistent with the deposition of layers, the rupture resulted from the generation

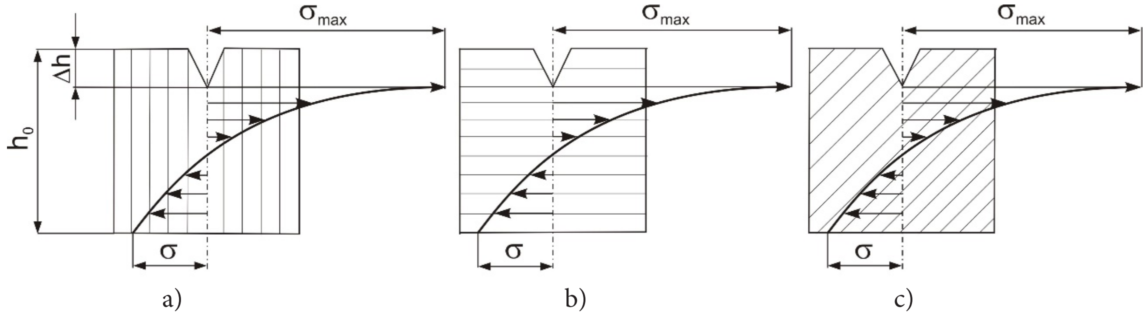


Fig. 8. Orientation of tensile stresses in relation to the direction of material layer build-up and the position of the notch during the failure (rupture) of the specimen: a) notch situated in parallel, b) notch situated perpendicularly (2) and c) notch situated at an angle of 45° in relation to the deposited layers (3)

of complementary contact stresses within layer boundaries, which led to the parallel shear-like displacement of those layers until the failure of the material (Fig. 8b). The areas characterised by the lowest structural strength were the boundaries between the successive layers of the specimen subjected to the SLM process. In terms of strength, the most favourable was the position of material layers at an angle of 45° in relation to the direction of tensile stresses (Fig. 8c).

The anisotropy of the toughness of specimens (identified in the tests) made using the SLM method and dependent on the position of the notch in relation to layers of the material necessitates the design of products taking into consideration the orientation of layers and the position of structural notches (cross-sectional changes) in relation to external loads (which could constitute areas of stress concentration).

The impact bend tests revealed a distinctly noticeable decrease in the toughness of the solid steel at a temperature of −20°C in comparison with that identified at a temperature of +20°C (Fig. 4). An increase in brittleness at lower temperature was a natural phenomenon. In turn, in the material subjected to selective laser melting it was possible to observe a slight decrease in toughness at the lower testing temperature (−20°C), regardless of the position of the notch in relation to the deposited layers. The reason for such material behaviour was the dual-phase ferritic-austenitic structure of the specimens obtained using the additive manufacturing method. Austenitic structures are characterised by low sensitivity to the reduction of temperature and transition into the brittle state, hence it was possible to observe a slight decrease in toughness at a temperature of −20°C.

The performance of stress relief annealing and the removal of internal stresses led to an increase in the tensile strength of the specimens obtained using the SLM method (Fig. 1). It was also possible to observe a slight increase in the plastic properties of the SLM specimens after heat treatment (Fig. 2).

The microstructural tests revealed significant differences between the structure of the specimens obtained using the additive manufacturing method and the initial structure of the solid steel (Fig. 5, 6). The structure observed in the SLM specimens was dual-phase, i.e. ferritic-austenitic. The use of large magnification revealed the presence of precipitated carbides of alloying elements, i.e. Ni and Cr (Fig. 6). Carbon bound in carbides precluded the obtainment of the martensitic structure. The presence of carbide phases (in the structure) precipitated along grain boundaries negatively affected toughness and favoured the formation of brittle fractures.

Summary

Leading SLM research centres observed that the most favourable operating conditions could be divided into factors directly affecting laser beam-related processes, i.e. power, laser beam travel rate, the distance between runs of the laser beam and into the remaining process parameters, i.e. layer thickness, the type of shielding gas, material and uniform layer deposition [1]. Tests discussed in publications [16-20] revealed the great importance of the appropriate composition of powder, i.e. the size of particles, the shape of particles characterised by low friction and high mobility as well as the fraction of fine particles filling cavities between large grains. In cases of additive manufacturing methods, the thickness of the melted layer should be twice as big as the largest grains of melted powder [21]. Other important factors include the number of exposures of a single layer and the distance between the number of scanning beam runs. The multiple exposure of the powder layer translates into the more favourable melting of the layer and the significantly lower porosity of fabricated elements [1]. The test results presented in publication [22] confirmed that a properly designed manufacturing process involving the use of the SLS/SLM methods and taking into account the above-named factors enabled the optimisation of the process and the fabrication of high-quality elements characterised by a density of near 100% and high mechanical properties. Research-related tests confirmed the high effectiveness of the above-named manufacturing methods without affecting the properties of the solid base material also in cases of titanium alloy Ti6Al4V or stainless austenitic steel 316L [23, 24].

The above-presented test results revealed that the properties of materials made using the additive manufacturing method (SLM) and metallic powder having the chemical composition corresponding to that of steel X5CrNiCuNb16-4 were significantly lower than those of the solid steel. The foregoing was primarily determined by the structure of the material matrix obtained after laser melting followed by heat treatment and the significant fraction of porosity in the volume of the printed element. The tests also revealed that the properties of the printed element and, consequently, finished products, depended on the direction of external load effect in relation to the orientation of deposited material layers. Under specific operating conditions, the lower mechanical properties of elements could be sufficient and satisfy related performance requirements. The method can also be used to fabricate prototypical elements of complex geometry and shape, which could subsequently be subjected to initial strength and operational tests before starting high-volume production under conventional manufacturing conditions.

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